# EXPERIMENTAL CHARACTERIZATION OF THE EFFECT OF CHARRING ON THE RESIDUAL LOAD CARRYING CAPACITY OF A STRUCTURAL FIBRE REINFORCED COMPOSITE

David J. Hill
Rimkus Consulting Group Inc.
Denver, Colorado 80237, USA &

José L. Torero
School of Engineering and Electronics
The University of Edinburgh
Edinburgh, EH9 3JL, UK

## ABSTRACT

An experimental study conducted to investigate the residual load carrying capacity of a commonly used structural composite plastic, isophthalic polyester, reinforced with S-glass fibreglass when exposed to heat-fluxes representative of a fire is presented. The purpose of this study is to explore the relationship between fire insult and the remaining flexural strength of a thermally damaged commonly used composite plastic. The samples were subjected to 20 kW/m² through 40 kW/m² heat fluxes for varying amounts of time. Selected samples were allowed to ignite to ensure that both radiant and fire exposures were considered. Resulting sample temperatures and mass loss quantities were recorded as a function of time through the use of implanted thermocouples and a load cell. All samples were allowed to cool in a zero moisture environment prior to being structurally loaded to failure using a 3-point bending machine. The results obtained tend to demonstrate a clear linear relationship between the depth of un-charred material (on a Non-Ignition sample) and its residual load carrying capacity. Furthermore, a linear correlation between the total amount of energy imposed on a sample and its residual strength is evident. The type of correlation depends on whether the sample ignited or not.

## INTRODUCTION

The construction industry has many uses for composite plastics and a myriad of types to choose from. Although much research has been conducted concerning the exposure of structural plastics to heat, very little has been done regarding the formation of a char layer and the remaining strength of a composite. Thus, the purpose of this study is to explore the relationship between fire insult and the remaining flexural strength of a thermally damaged commonly used composite plastic.

Most studies dealing with composites concentrate on such topics as flammability, heat release rate, ignition, and the smoke and toxic gas emissions of a polyester resin based composite material[1-3]. Many studies differed from one and another through the use of varying types of resins, varying types of reinforcements, different reinforcement alignments and the use of additives to increase fire retardancy.

Davies and Wang[4] analyzed the transient thermal response of glass-fiber reinforced polyester composites exposed to cellulosic and hydrocarbon modeled fires. They were able to generate a model that calculated temperature profiles, which were in good agreement with experimental data for these fire types but cautioned that discrepancies did occur as the model was unable to account for all physical processes that occurred when the material was heated. A common theme among many

studies is the comparison of the properties of different types of resins. Gibson and Hume[5] compared the heat release rate, ignitability, smoke emission and fire resistance of polyester, vinyl ester, epoxy and phenolic laminates. The research showed that the phenolic resin had the most favorable results. Yet phenolic resin composites are not widely used due to the cost and complexity of manufacture. Another comparison done was that by Mouritz and Mathys[6], which examined the post-fire properties of glass-reinforced polyester, vinyl ester phenolic composites. Despite the superior fire resistance of phenolic resin, it was found that the post-fire tension and flexural properties of all resins used were similar. The tests done by Mouritz and Mathys differed from the ones done for this work in that the type and alignment of the reinforcement, the dimensions of the samples and the flexural testing method (four-point bend method) were not the same. Furthermore, the work of Mouritz and Mathys did not look at the any correlations between strength and char depth.

Only the work of Pering, Farrell and Springer could be found that had any dealings with the formation of a char layer [7]. This study concentrated on a single material made of the most widely used type of composite plastic resin in conjunction with a commonly used type of reinforcement in a simple alignment. A baseline study was conducted and looked for a correlation between char layer depth and the residual strength of this material. Despite the fact that this study showed that a rapid decrease of shear and tensile strengths and moduli could be correlated to the char thickness no follow-up studies could be found in the literature.

For this study a cone heater and a Universal Testing Machine setup for three-point testing was used to test hand made composites (isophthalic polyester, reinforced with S-glass fibreglass). The composite was first heated and charred to varying degrees by modifying exposure duration as well as exposure intensity. These samples were then allowed to cool and were flexurally loaded until failure occurred. The depth of the char layer due to the sample heating was then analyzed in relation to the residual flexural strength of the samples.

## EXPERIMENTAL APPARATUS AND PROCEDURES

The experimental process consisted of several different stages including manufacture, determining the thermal properties of the material, the charring of the samples and the structural loading of these burnt samples in a three-point bending machine. The isophthalic polyester composites that were used for the testing were made following a given blueprint[8] and contained unidirectional S-glass fiberglass as reinforcement.

The plastics used in this research were made to simulate a polyester resin with S-glass fiberglass reinforcement composites and were manufactured using the hand-laid technique. Similar procedures have been used at NIST by Chin and co-workers[5]. Isophthalic polyester resin / S-glass composites were produced from moulds of dimensions 200 mm x 250 mm x 12.5 mm then cut using a *Bridgeport* mill and a wet cutting diamond tile blade to samples of 40 mm x 250 mm x 12.5 mm. Cutting several samples from the same mould ensured similar properties existed per group. Thermocouples were included in the construction of them. Although variances might be present within batches or the use of thermocouples might have some effect on the resistance of the material, every effort was made to obtain homogeneous samples.

Thermocouples were positioned so that the composite sample had a thermocouple on the bottom and top faces as well as one located in the mid section of the composite. All thermocouples were of OMEGA “K” type 0.1 mm in diameter. The thin thermocouples were selected so that the strength of the composite would not be severely altered with it running through the samples. The top thermocouples were fastened to the sample by drilling a small hole on the surface, inserting the thermocouple, and then pouring resin to fill the hole. The mid thermocouples were included in the manufacture of the composites and the bottom thermocouples were attached to the bottom of all samples using tape. Every effort was made to position the thermocouple on the geometrical centre of the plane of the composite. A series of tests was conducted as a reference with no thermocouples

showing no difference in the results. Further details on the manufacture of the composites can be found in reference [9].

The composites, after been made and cut, were exposed to varying degrees of radiant heat for several time periods. It was these samples that the bulk of the research focused on. The heating of the samples was done under a cone heater following ASTM E-1354[10] and the resulting temperature profiles of the surface, interior, and unexposed underside of the composites were recorded to develop a heat penetration versus time profile. In addition, the mass lost due to pyrolysis versus time was also logged using a load cell. All data collected was done using a FLUKE© data acquisition system. To ensure that the entire length of a sample did not get exposed to the heat fluxes, the ends of each composite sample were wrapped in aluminium foil so that only a centre portion (approx. 100 mm) was left uncovered. Heat flux measurements confirmed that the cone heater provided a constant heat flux over the exposed surface with a variation from the mean value that do not exceed +/-3.5%.

After allowing the samples to reach room temperature again in a zero humidity environment, the samples were then flexurally loaded using a Universal Testing Machine (MTS Systems Corporation) that determined the remaining strength of the composites when subjected to three-point bending. Tests were conducted using the ASTM D-790[11] standard procedures. The loading rate used was 0.51cm per minute and was selected as a quick loading rate to minimize creep development. The samples were loaded until a maximum load carried had been reached. This entailed watching the loading results and when it became evident that a sample could no longer hold large loads due to too much damage, the tests were stopped. Failure is defined as a sudden decrease of the load on the nose following displacement.

Once the flexural testing was complete, the char depth per sample was measured using a micrometer. Two separate methods were used to determine the depth of char, both using a micrometer. For the first method, the micrometer was opened so that the small end protruded at least 10 mm out. This end was then inserted in to the char layer so that the micrometer was perpendicular to the sample's surface. Insertion of the end meant forcing it in until solid composite material underneath was reached. Then the micrometer was closed slowly so that the face perpendicular to the small end was just above the char layer. This measurement was then recorded as the char layer "fiber penetration" measurement. The second method used in estimating the char layer was to examine the side of the sample and measure how far down the char layer had reached, again with the micrometer. This depth on all data is referred to as the "char layer" measurement. Both measurements showed consistent results for all tests.

## RESULTS

### Ignition Delay Time

Composite samples were used to establish the ignition delay time as a function of exposure. This was necessary as the heat flux imposed on a sample due to it having ignited is severely modified by the heat feedback from the flame. With that in mind, there are obviously two characteristic regions of study, ignited and non-ignited samples. A series of smaller samples, 100 mm x 100 mm were subjected to varying degrees of heat fluxes to estimate the ignition delay time of the material. The resulting plot was used to plan what type of exposure the full sized samples would receive. The results of these tests are presented in Figure 1.

Figure 1 shows the evolution of the ignition delay time with the heat flux. Exposures to the right of this curve will result in ignition. The plot also presents the exposure intensities as well as durations were used for the full sized samples (stars). The choice of testing conditions guaranteed that ignition and non-ignition tests were considered.

Figure 1 Ignition delay time as a function of heat exposure. The stars represent conditions chosen for charring tests.

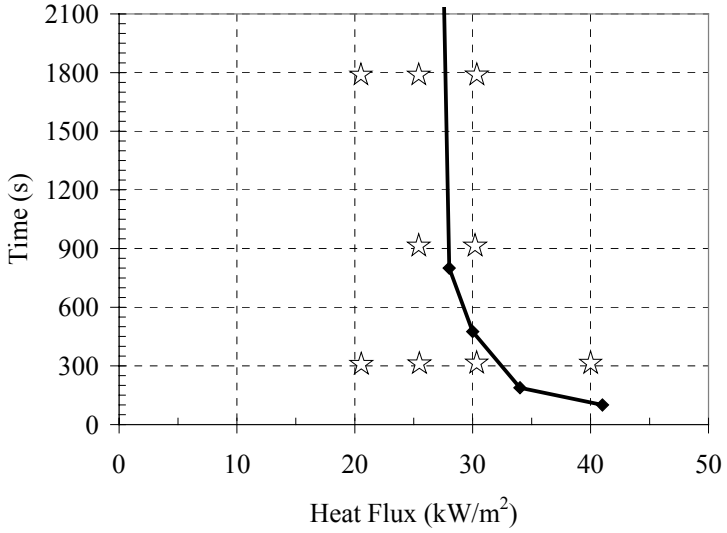
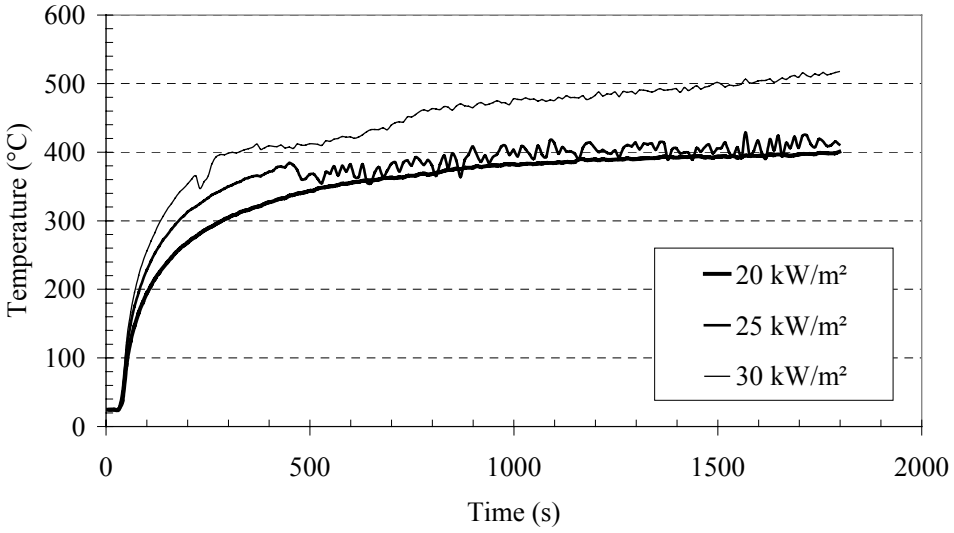



Figure 2 presents the evolution of the surface temperature with time. It can be observed that the temperature increases until pyrolysis of the resin is initiated. At this point gasification of the resin occurs at an almost constant temperature (400°C). This is an important measurement since it indicates that throughout the gasification process the surface temperature can be assumed to be constant. The obvious discrepancy between the 30 kW/m$^2$ data and the other temperature histories is a common occurrence resulting from the separation of the thermocouple from the surface.

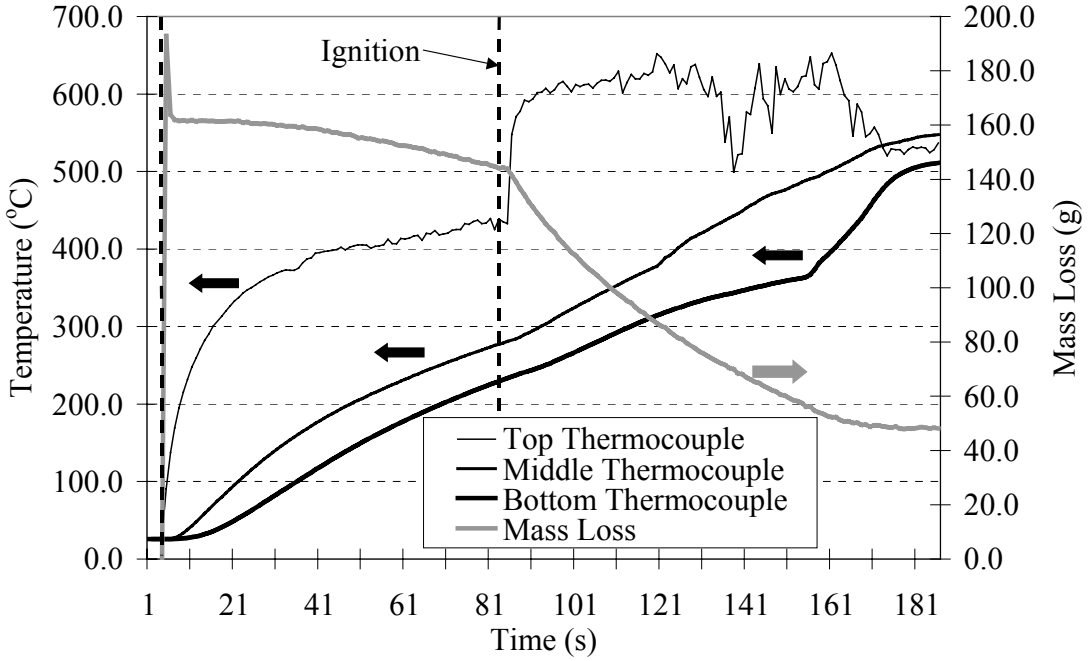
Figure 2 Surface temperature as a function of time




The initial temperature and mass loss versus time data offered nothing remarkable. As the exposure duration and intensity were increased from one group to the next, temperatures and the quantities mass loss increased as well. The rate of mass loss changed, regardless of exposure, due to the ignition of the sample. Figure 3 represents one such sample. From the plot, several things are to be noted, after a preheat region (approximately 40 seconds) there is an apparent linearity of the mass loss until ignition occurs. Then, the mass loss follows a potentially exponential decay after ignition. It is important to notice a sudden increase in temperatures due to the ignition. The middle and bottom thermocouples follow similar trends but the changes are less dramatic. The bottom thermocouple shows a temperature

increase almost immediately after the sample was exposed to the heat flux, this indicates that heat losses to the back of the sample will have to be included in any thermal analysis of the process.

Figure 3 Temperature and Mass Loss vs. Time plot for a sample that ignited (32.5 kW/m$^2$)




**Flexural Testing**

The flexural loading of any beam puts the top portion of it in compression, and the bottom portion in tension. The neutral axis separates these portions and feels no physical deformation. With a composite plastic, the fibers in the top portion crowds together within the resin and this combination is the resisting force to compression. On the bottom, the resin transfers tension to the fibers whose modulus of elasticity is greater than that of the resin. The maximum load that a beam can support then becomes a function of both the compressive and tensile strengths of the beam. Should one side start to fail, the slack is re-proportioned between the tension and compression sections of the beam. This shifting of forces coincides with an adjustment of the location of the neutral axis and may unduly burden either the tension or compression segments and lead to failure.

In the three-point bending tests done, the samples were simply supported and developed reactions normal to the roller supports as well as to the central roller applying the load. Being that all supports were rollers, the ends of the beam were able to rotate freely and thus could not develop moment couples. The beam was intended to overhang each support by 10% of its design length thus ensuring it remained supported as it deflected. Under these assumptions the equation that solves for the maximum stress in the outer surface of the samples supported as such, is as follows:

$$\sigma_f = \frac{3Pl}{2wt^2} \qquad [1]$$

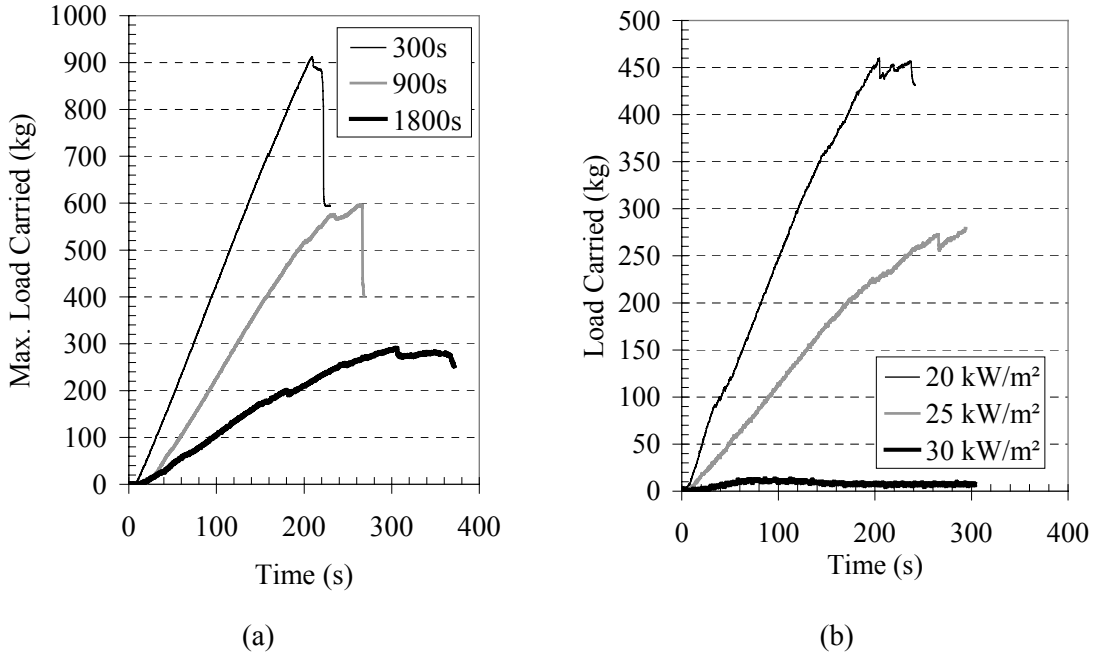
Where $\sigma_f$ is the maximum stress, "P" the applied load, "l" the distance between bottom supports, "w" the width of the sample and "t" the thickness of the sample. For the testing, the distance between the bottom supports remained constant at 220 mm and all samples were cut so that they had a width of 40 mm. The thickness of the sample obviously depended on the manufacture of the sample but 12.5 mm was the intended thickness; most composites were just slightly larger than this. The composite material is considered to be elastic.

For the testing, the support span to thickness ratio was the important factor. For ASTM D-790, a minimum support span-to-thickness ratio of 16:1 was required. A large span to thickness ratio such as this is used to ensure the specimen fails flexurally. It is also used to minimize inter-laminar shear stresses between the plies. Because the samples were charred to varying degrees, as char depth increased, the effective depth of the samples shrank. This meant that the support span to depth ratio would increase.

All samples produced cracking noises when tested. The difference between the samples was that when they started emitting these sounds, the frequency of the cracking and the intensity of the noise were dissimilar for the different groups. Stronger samples tended to get louder as testing continued; most culminating with a sudden intense crack that represented the maximum load had been reached. Samples of reduced strength also produced cracking noises but their intensity was noticeably diminished. They tended not to have a final large cracking noise, rather a series of smaller groups of sounds. These sounds represent fibers in the samples failing in tension. Although all samples were tested until the maximum load was reached, all samples still retained some strength as none of the samples clearly broke in to two. This demonstrated that although a significant portion of fibers in the composite had failed, not all fibers were damaged.

From the structural testing, load versus time, time to failure and maximum deflection values were recorded. Analysis for each test and then data comparison was done as needed. Figure 4 presents some examples of the raw data. It becomes apparent when observing Figure 4 that both exposure duration (4(a)) and exposure intensity (4(b)) would affect the flexural strength of the composites. As both the exposure duration and intensity increased independently, the strength of the samples decreased accordingly. Although the plots only illustrate data from a few samples, the other samples were similar in result. In comparison to these outcomes, the samples made of resin only demonstrated very little flexural strength which showed how in tension, the fibers in the composite retained the brunt of the stress.

Figure 4 Examples of flexural strength test results showing the evolution of the flexural strength with (a) exposure time (25 kW/m$^2$) and (b) heat flux (1800 sec)




In addition to the previously mentioned, it was found that all plots of samples placed under the cone heater for 300s had linear strength vs. time lines, regardless of heat flux. As the time interval

increased to 900s and then 1800s, the linearity of the plots decreased markedly. Another trend noticed was that by preventing ignition but with the same heat exposure, the strength of a sample was noticeably increased. The difference in strength from Non-ignition to Ignition averaged out to be a 66% reduction. A final trend observed in the flexural strength data was how in most cases, the weakest sample of a group was the one containing the middle thermocouple. The reduction in strength was not of drastic proportions but certainly evident in the data.

There seems to exist a relationship between the strength of the samples and the mass lost Pering, Farrell and Springer[4] also found that correlations exist between the flexural strength of samples and the amount of char but it should be noted that their experimental set-up as well as the type of material being tested were different from that of this research. The examination of Ignition sample mass loss data did not present any clear correlations. Nevertheless when the char depth (Fibre Penetration) is presented as a function of the total energy input (Figure 5) a linear dependency emerges. For ignited samples the correlation seems quite good despite the fact that ignition adds a significant heat input. The fact that ignited samples were only allowed to burn for a very short period after ignition (Figure 1) might explain this observation. For non-ignited samples the correlation is not as good but it is still present. For the lower heat fluxes the surface might have reached thermal equilibrium resulting in significant periods of exposure with no further charring. A clear distinction between ignited and non-ignited samples appears.

Figure 5 Fiber penetration as a function of the total energy input (heat flux multiplied by time of exposure). (I) represent ignited samples and (NI) non-ignited.

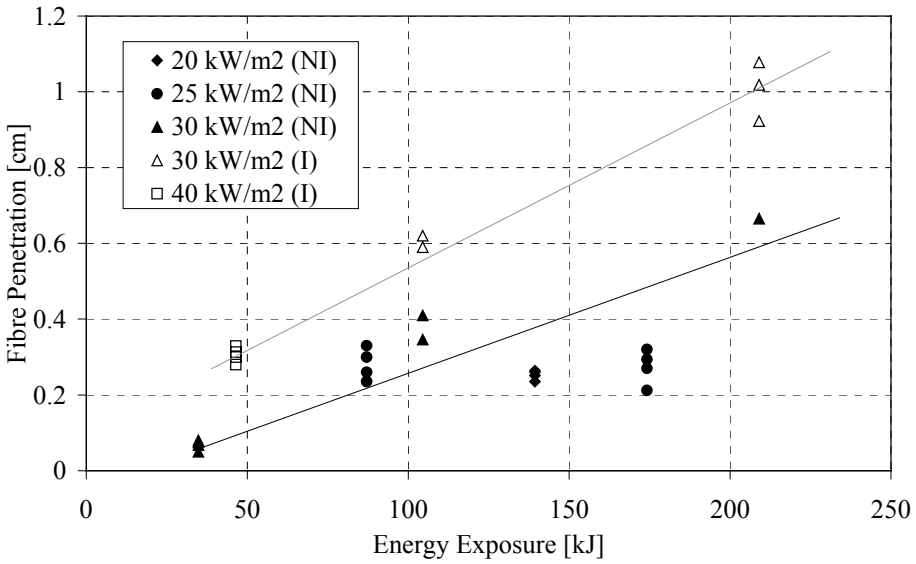
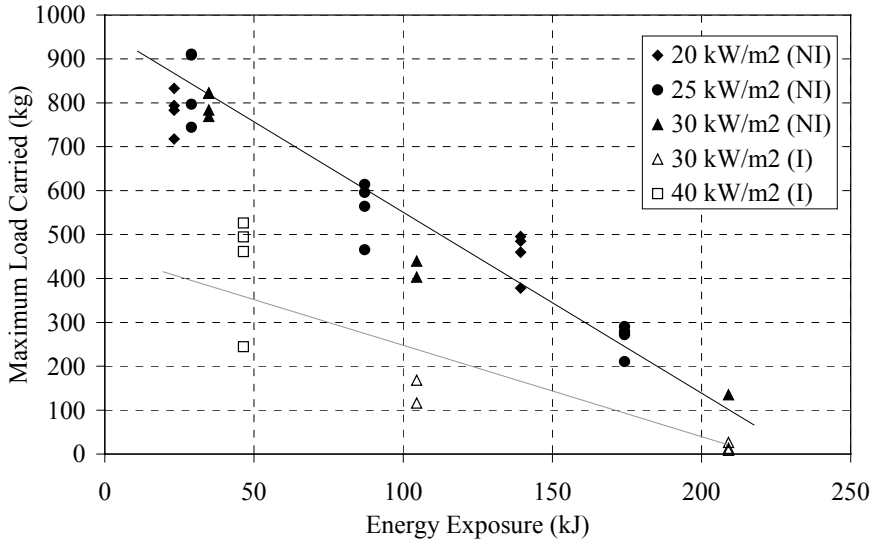


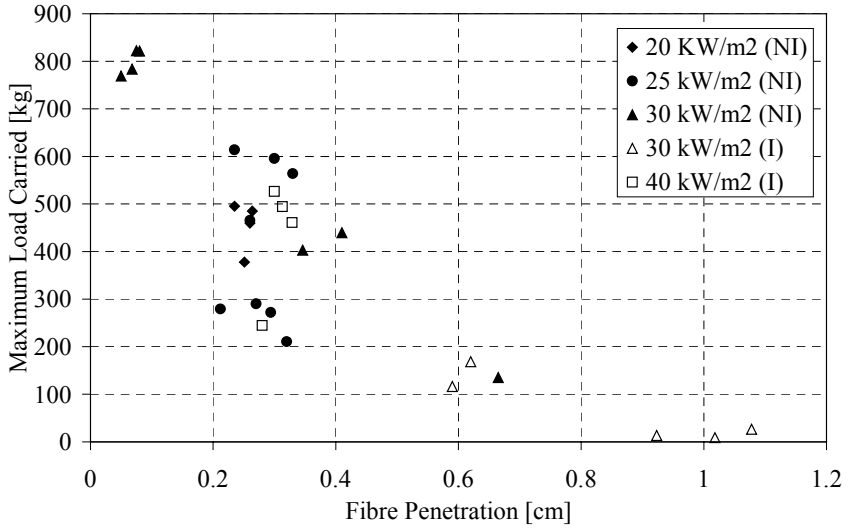

Figure 6 presents the maximum carried load as a function of the total energy exposure. Similar trends to those observed in Figure 5 appear. The maximum load carried decreases linearly with the exposure and a clear distinction appears between non-ignited and ignited samples. In this case the scattering of the data is more evident for the high heat fluxes and ignited samples. This indicates that during the flaming period the heat flux might not generate further char penetration but does have a significant effect on the material strength. Figure 3 shows that post-ignition there is a large mass loss and a significant temperature increase in-depth. These factors can definitely contribute to the above observations.

Figure 6 Maximum Load Carried as a function of the total energy input (heat flux multiplied by time of exposure). (I) represent ignited samples and (NI) non-ignited.




Combining Figures 5 and 6 the maximum load carried can be presented as a function of the char depth. The data collapses for both ignition and non-ignition tests showing that the main parameter controlling the maximum load carried is the residual non-charred material. These observations confirm those of reference [1] and provide a clear indication that the maximum load carried of a composites can be modelled as a function of the charring rates of the material.

Figure 7 Maximum Load Carried as a function of the fiber penetration. (I) represent ignited samples and (NI) non-ignited.




The data scattered in Figure 7 together with the non-linearity of Load vs. Time graphs of samples whose exposure duration were greater than 300 seconds, reveals that there exists areas of reduced strength that are not necessarily charred. These layers of thermal damage must possess less than full strength, as their plots were not linear. If there only existed two zones - char damage and undamaged composites, all Load vs. Time plots would be linear until failure.

## Conclusions

An experimental study conducted to investigate the residual load carrying capacity of a commonly used structural composite plastic, isophthalic polyester, reinforced with S-glass fibreglass when exposed to heat-fluxes representative of a fire has been presented. The results obtained tend to demonstrate a clear linear relationship between the depth of un-charred material (on a Non-Ignition sample) and its residual load carrying capacity. Rate of mass loss once ignition has occurred, is related to the rate of combustion. The fire on the sample radiates heat back to the material undergoing thermal decomposition and thus as more energy radiated back, the more volatiles are released. Therefore the mass loss is significantly larger for Ignition samples. Nevertheless, the linear relationship between the depth of un-charred material and its residual load carrying capacity remains.

The important parameters that influence this strength come from the matrix (resin), the reinforcing fibres and the interface between the two previously mentioned. S-glass fibreglass is known as the strongest type of glass fibres commercially available and yet its strength can mean very little should the bond that links the resin to this reinforcement be weak. For short exposures the samples can be divided into charred and un-degraded regions. In the un-degraded region the bond between the fibres and matrix remains unaffected. For extended exposure it could be observed that there exist areas of reduced strength that are not necessarily charred. These layers of thermal damage with less than full strength are revealed by non-linear Load vs. Time graphs and the data scatter. As a footnote, increased temperatures inside the samples do not damage the strength of the fibreglass as it has a melting point above 1000°C.

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